PB86-231081

Analytical Methods for Determination of Selected Principal Organic Hazardous Constituents in Combustion Products

Southern Research Inst., Birmingham, AL

Prepared for

Environmental Protection Agency
Research Triagle Park, NC

1986

U.S. Department of Commerce
National Technical Information Service
NTIS

PB86-231081

TECHNICAL REPORT DATA
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1. REPORT NO. EPA/600/J-86/116	2.
3. RECIPIENT'S ACCESSION NO.	
4. TITLE AND SUBTITLE: Analytical Methods for Determination of Selected Principal Organic Hazardous Constituents in Combustion Products (Journal Version)	5. REPORT DATE: 1986
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S): R. E. Adams, R. H. James, L. A. Burford, and H. C. Miller (SoRI); and L. D. Johnson (EPA/AEERL)	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Southern Research Institute, 2000 Ninth Avenue South, Birmingham, Alabama 35255-5305	10. PROGRAM ELEMENT NO.
	11. CONTRACT/GRANT NO. 68-02-3696
12. SPONSORING AGENCY NAME AND ADDRESS: EPA, Office of Research and Development, Air and Energy Engineering Research Laboratory, Research Triangle Park, NC 27711	13. TYPE OF REPORT AND PERIOD COVERED: Journal article; 6/83-2/84
	14. SPONSORING AGENCY CODE: EPA/600/13

15. SUPPLEMENTARY NOTES: AEERL project officer is Larry D. Johnson, Mail Drop 62, 919/541-7943. Environ. Sci. Technol., Vol. 20, No. 7, 1986, pp 711-716.

16. ABSTRACT: The paper gives an overview of generalized analytical procedures for determining designated principal organic hazardous constituents (POHCs) in combustion products; it also gives some examples of the techniques used for compounds such as 2,3,7,8-tetrachlorodibenzodioxin, selenourea, and several organo-arsenicals. Emissions from hazardous-waste combustion must be monitored to determine the destruction removal efficiency (DRE) for each designated POHC. Analytical methods for more than 150 POHCs have been reviewed. A generalized HRGC/LRMS method to determine volatile, thermally stable POHCs has been developed. A method based on HPLC with UV detection has also been developed to provide an alternative for determining nonvolatile or thermally labile compounds. The generalized methods are applicable to many compounds, but specific POHCs may require variations in GC/MS or HPLC procedures. Inclusion in the paper does not mean that the sampling or analysis method is an official EPA method. Official test methods for hazardous waste related programs are published in SW-846, Test Methods for Evaluating Solid Waste, as well as in the Federal Register.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pollution	Pollution Control	13B
Combustion Products	Stationary Sources	21B
Toxicity	Principal Organic Hazardous Constituents	06T
Sampling		14B
Analyzing	Destruction Removal Efficiency	
Organic Compounds		07C

18. DISTRIBUTION STATEMENT: Release to Public	19. SECURITY CLASS (This Report): Unclassified	21. NO. OF PAGES
	20. SECURITY CLASS (This page): Unclassified	22. PRICE

EPA Form 2220-1 (9-73)

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Environ. Sci. Technol. 1986, 20, 711-716

EPA/600/J-86/116
JOURNAL ARTICLE

Analytical Methods for Determination of Selected Principal Organic Hazardous Constituents in Combustion Products

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■ The emissions from hazardous-waste combustion must be monitored to determine the destruction removal efficiency (DRE) for each designated principal organic hazardous constituent (POHC). Analytical methodology for more than 150 POHCs has been reviewed. A generalized high-resolution gas chromatography/low-resolution mass spectrometry (HRGC/LRMS) method to determine volatile, thermally stable POHCs has been developed. A method based on high-performance liquid chromatography (HPLC) with ultraviolet (UV) detection has also been developed to provide an alternative for the determination of nonvolative or thermally labile compounds. The generalized methods are applicable to many compounds, but specific POHCs may require variations in gas chromatography/mass spectrometry (GC/MS) or HPLC procedures. This paper will present an overview of the generalized procedures as well as some examples of the techniques used for compounds such as 2,3,7,8-tetrachlorodibenzodioxin, selenourea, and several organoarsenicals.

Introduction

Background. As part of the Resource Conservation and Recovery Act (RCRA) (*1*), the U.S. Environmental Protection Agency (EPA) has promulgated proposed, interim, and final regulations for the owners and operators of facilities that treat hazardous wastes by incineration. The purpose of the regulations is to ensure that such incinerators are operated in an environmentally responsible manner. The Consolidated Permit Regulations (Parts 122–125, 40 CFR) (*2*) cover a range of activities, including operational performance standards, waste analysis, trial burns, monitoring and inspections, record keeping and reporting, and the establishment of emission-control criteria. The specific details for each incinerator facility are authorized by facility permits.

An important criterion upon which all operational specifications are based is the destruction and removal efficiency (DRE) of the incinerator. This value, which is defined in terms of the levels of the potentially hazardous substances in the waste feed and stack gas, must be greater than 99.99% for proper incineration. A list of potential "principal *organic* hazardous constituents" (POHCs) is found in Appendix VIII, Part 261, 40 CFR (*3*). The list includes organic, organometallic, and inorganic compounds. Organic POHCs may be selected from this list.

The overall strategy for waste characterization includes test procedures to determine the characteristics of the waste and analytical procedures to determine the composition of the waste. The manual, "Sampling and Analysis Methods for Hazardous Waste Combustion" (*4*), includes test procedures for proximate, survey, and directed analysis. An overview of this analytical approach is shown in Figure 1. The brief discussion here is limited to directed analysis. Modification of analytical methods applicable to the identification and quantification of POHCs is now of primary interest to us.

The directed analysis portion of the waste-characterization scheme provides qualitative confirmation of compound identity and quantitative data for the potentially hazardous constituents that might reasonably be expected to be present in the waste, based on engineering judgment and on the results of proximate and survey analyses. Directed analysis does not involve the screening of every waste sample against the complete hazardous-component list. Directed analysis consists of the minimum set of analytical techniques that can be applied to the waste for qualitative identification and quantitative determination of the components that are actually present.

In practice, the results of the directed analysis will establish whether the waste contains the suspected pollutant and will establish the concentration range at which the pollutant may be expected to be found. Directed analysis will also be used to confirm and quantify any unexpected hazardous components not identified in the survey analysis. These data are essential for selection of the appropriate POHCs to be monitored.

Purpose. Approximately 400 compounds are included in Appendix VIII, Part 261, 40 CFR. Previous work (*5*, *6*) involved the evaluation of generalized GC/flame ionization detection (FID), GC/MS, and HPLC/UV methods for the determination of approximately 150 compounds from this list of POHCs. However, the survey analysis portion of a waste-characterization scheme often specifies compounds for determination in incinerator effluent that are not amenable to previously developed methods (*5*, *6*). Therefore, this current research involved the development of specific GC/FID, GC/MS, and HPLC/UV methods for the determination of several of these compounds. This paper presents preliminary data on methodology for two groups of compounds, organometallics and chlorinated dibenzodioxins.

Organometallic compounds such as benzenearsonic acid, hydroxydimethylarsine oxide (cacodylic acid), phenylmercuric acetate, selenourea, and tetraethyllead were selected as candidate POHCs for this study. These compounds were chosen from Appendix VII, Part 261, 40 CFR (*3*), because of expected difficulties in analyzing these compounds by previously developed methods. Tetraethyllead was easily measured by the generalized GC/FID and GC/MS procedures. Selenourea required a new HPLC/UV option. The two organoarsenic compounds and the organomercury compound could not be determined by HPLC/UV with adequate sensitivity and were not easily determined by GC.

Volatile hydride generation has been demonstrated to be useful for the determination of inorganic and organic arsenic compounds (*7*). Also, Soderquist et al. (*8*) has demonstrated that hydroiodic acid (HI) will convert nonvolatile hydroxydimethylarsine oxide to volatile iododimethylarsine. This paper demonstrates that iodide derivatives of benzenearsonic acid, hydroxydimethylarsine oxide, and phenylmercuric acetate can be determined by GC/FID and GC/MS.

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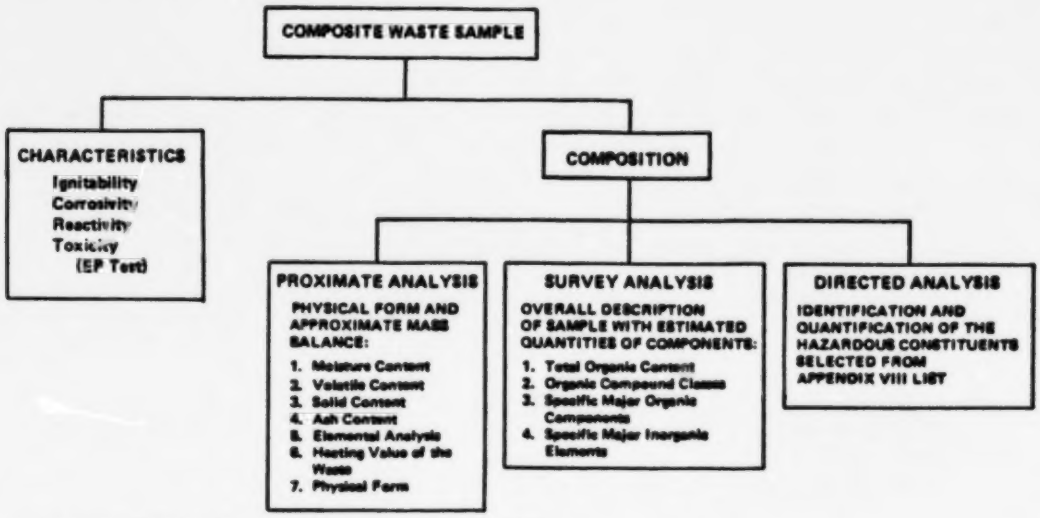


Figure 1. Overview of the analytical approach for waste characterization.

The determination of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) in water, soils and sediment, and incinerators (*9–14*) has been of increasing interest. These methods use isomer-specific high-resolution (HR) capillary GC columns and selected-ion-monitoring (SIM) low-resolution (LR) MS. This paper compares the generalized GC/MS procedure developed in earlier work (*5, 6*) with the more selective and sensitive procedures developed by the EPA.

Experimental Section

Materials. 2,3,7,8-TCDD was obtained as a 7.97 μg/mL solution from the EPA. [$^{13}C_{12}$]-2,3,7,8-TCDD and [$^{37}Cl_4$]-2,3,7,8-TCDD were obtained from Cambridge Isotope Laboratories. Tetraethyllead was obtained from Ethyl Corp. Hydroiodic acid (Kodak) was a 55% (w/v) certified grade without stabilizer. Hydroxydimethylarsine oxide (cacodylic acid) was obtained from Chem Services, Inc. Selenourea, benzenearsonic acid, and phenylmercuric acetate were obtained from Aldrich Chemicals. All other chemicals were reagent grade or better.

Equipment. The GC/FID work was performed on a Hewlett-Packard Model 5840 GC. GC/MS work was performed on a Hewlett-Packard Model 5985A GC/MS. Both instruments were equipped for split-splitless injections on capillary columns. The HPLC/UV work was performed on a Hewlett-Packard Model 1084B HPLC with a variable wavelength UV–vis detector (190–600 nm) and an automatic sampling system.

Procedures. The generalized GC/MS and GC/FID conditions are the following: instruments—HP5985A GC/MS, HP5840 GC/FID; columns—two matched SE-54 bonded-fused silica capillary columns, 0.32 mm i.d., 25-m length; carrier gas—helium, 30 cm/s; injection—2 μL splitless; column temperature program—40–280 °C at 10 °C/min, hold 15 min; injection temperature—290 °C; FID temperature—300 °C; MS parameters—scan 41–450 amu/s, electron ionization at 70 eV, source temperature set to 200 °C, capillary column plumbed directly into the ion source. The initial operating conditions chosen were a synthesis of conditions that would allow the separation and detection of as many compounds as possible. Modifications of temperatures and rates may be necessary or desirable to optimize the analysis for specific compounds.

The selected-ion-monitoring GC/MS conditions for 2,3,7,8-TCDD are the following: instrument—HP5985A GC/MS; column—Chrompack CP-SIL-88 fused silica capillary, 0.23 mm i.d., 60-m length; carrier gas—helium, 30 cm/s; injection—3 μL splitless; column temperature program—45 °C for 3 min, to 190 °C at 25 °C/min, to 240 °C at 5 °C/min, hold; injection temperature—290 °C; MS parameters—selected ions *m/z* 257, 320, 322, 328, 332, and 334 for 70 ms each, source temperature set to 200 °C, capillary column plumbed directly into the ion source.

The HPLC/UV conditions are the following: instrument—HP 1084B with a variable wavelength UV–vis detector; column—Waters μBondapak-CN, 10-μm particle size, 3.9 mm i.d., 30-cm length; solvents—A = 1% acetic acid in distilled deionized water, B = acetonitrile; gradient program—10% B for 5 min, 10–100% B in 30 min, hold at 100% B for 5 min, flow rate of 1 mL/min.

Sample Preparation: Sorbent Tubes. Sorbent tubes were prepared by packing 4 mm i.d. glass tubes with ≃7.5 cm (400 mg) of XAD-2 resin (Applied Science, Residue Free, 20/50 mesh) and with silanized glass-wool plugs, front and back (Figure 2). These tubes were spiked (in triplicate) with aliquots of the organometallics in methanol. The first set of tubes was allowed to air-dry for ≃1 h before extracting with two 5-mL portions of methanol. The methanol extracts were combined in 50-mL centrifuge tubes and blown dry with a gentle stream of N_2 which had been passed through a charcoal filter. The residues were shaken with 1 mL of HI for 30 s, and the derivatized organometallics were extracted into 10 mL of hexane. A blank (unspiked) XAD-2 sorbent tube was carried through the procedure with the spiked tubes.

Another set of tubes was packed, spiked, and allowed to sit for 3 days. The tubes were then extracted, and the extract was derivatized and analyzed in the same manner as above.

The last set of sorbent tubes was packed, spiked, and then allowed to sit for 1 h. Warm, moist air (≃85% relative humidity) was then pulled through the tubes for 2 h at a rate of ≃200 mL/min. A flask was placed at the exit end of the sorbent tube to trap condensate. The condensate in the flask was also analyzed for the organometallics.

Filter Samples. Glass-fiber filters (Gelman Type A/E) were spiked in triplicate with 1-mL aliquots of a methanol solution of the organometallics (≃1 mg/mL). Filters were allowed to air-dry. Extraction of the filters with methanol was carried out in 200-mL screw-capped centrifuge bottles.

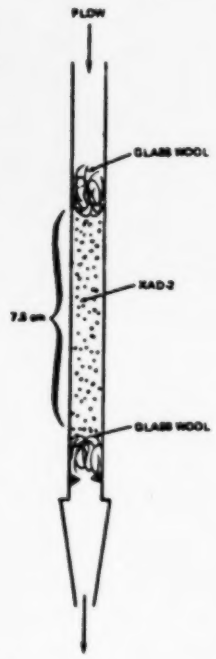


Figure 2. XAD-2 sorbent tube.

The first triplicate set was extracted and analyzed the same day of spiking. Filters were extracted with two 10-mL portions of methanol shaking on a gyratory shaker for 10 min. The methanol extracts were dried with a gentle stream of N_2. The residues were shaken with 1 mL of HI for 30 s. The derivatized organometallics were extracted into 10 mL of hexane. A blank filter was carried through the procedure with the spiked filters. Another triplicate set of filters was spiked, allowed to sit for 3 days, and then analyzed in the same manner.

Water Samples. Deionized water (50 mL) was spiked with 1-mg quantities of the organometallics. A 1-mL aliquot of HI was added to one sample and the sample shaken for 30 s. The derivatized water sample was then extracted with 20 mL of hexane. Another spiked water sample (50 mL) was dried to a residue using a gentle stream of N_2. This residue was then derivatized with a 1-mL aliquot of HI. The derivatized organometallics were then extracted into 20 mL of hexane.

Quality Control

The GC/FID, GC/MS, and HPLC/UV methods were calibrated with standard solutions of POHCs. For each POHC in the study, a four- or eight-point calibration curve was generated. For GC/FID and GC/MS determinations, relative responses were used to give calibration curves. Relative response was calculated as a ratio of detector responses for the standard POHC to that of the internal standard, anthracene-d_{10}. The HPLC/UV method was calibrated by an external standard technique. Internal standardization of the HPLC/UV method was not used because a single internal standard that was appropriate for all POHCs from Appendix VIII, 40 CFR, Part 261 (*3*), requiring HPLC/UV analyses could not be selected. Also, a linear-regression equation and correlation coefficient from the calibration data were calculated. Typically, correlation coefficients are in excess of 0.9900. The calibration curve can be used to estimate the sensitivity expected in the determination of a particular analyte. Also, each curve demonstrates the linearity of the detector response within the range of quantities investigated.

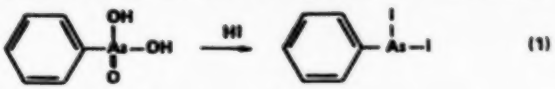
The precision of the determinations by GC/FID and GC/MS was assessed by triplicate injections of a standard solution of each POHC. However, for HPLC/UV determinations, five replicate injections were made. The estimate of precision of the replicate determinations is expressed as the relative standard deviation (RSD). The range of RSD for GC/FID is 1–4%. The range of RSD for GC/MS is 2–9%. The RSD for HPLC/UV determinations is less than 5%.

Any laboratory that participates in the analysis of POHCs will likely follow a formal quality control program. The relative retention times, on-column detection limits, chromatograms, calibration curves, ion relative abundances, mass spectra, and relative standard deviations in this paper will be a guide for other analytical laboratories. Because of subtle differences among laboratories and analytical instrumentation, laboratories should generate data that will establish formal criteria for the analysis of POHCs.

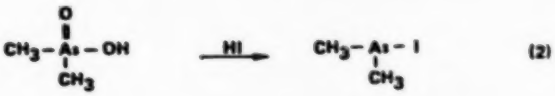
Results and Discussion

Organometallics. The five organometallic compounds targeted required three different methods of analysis. The general GC/FID and GC/MS methods were used to determine tetraethyllead. Benzenearsonic acid, hydroxydimethylarsine oxide, and phenylmercuric acetate were derivatized with hydroiodic acid to improve thermal stability and volatility and determined by GC/FID and GC/MS. Selenourea and benzenearsonic acid were determined by HPLC/UV.

The hydroiodic acid derivatization procedure is similar to the procedure used by Soderquist et al. (*8*). Equations 1–3 describe the derivatization process for the three organometallics. The reactions appear to proceed rapidly and quantitatively. The general GC/FID and GC/MS procedures were used to determine the derivatives.



(1)



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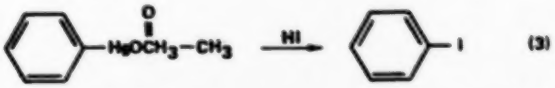

(3)

In Table I the GC/FID and GC/MS relative retention times and estimated on-column detection limits for benzenearsonic acid, hydroxydimethylarsine oxide, phenylmercuric acetate, tetraethyllead, and 2,3,7,8-TCDD are presented. The on-column detection limit is the quantity of each analyte that was estimated to give a detector response of twice the background signal. Typical values ranged from 0.7 to 11.8 ng for GC/FID and from 0.8 to 3.8 ng for GC/MS. The absolute intensity of the base peak in the mass spectrum was used to develop calibration curves. The precision of replicate determinations for GC/FID and GC/MS is presented in Table II. The mass spectra of the HI derivatives of benzenearsonic acid, hydroxydimethylarsine oxide, and phenylmercuric acetate are presented in Figure 3A–C.

HPLC/UV. In Table III the retention times, on-column detection limits, and wavelengths of detection of the

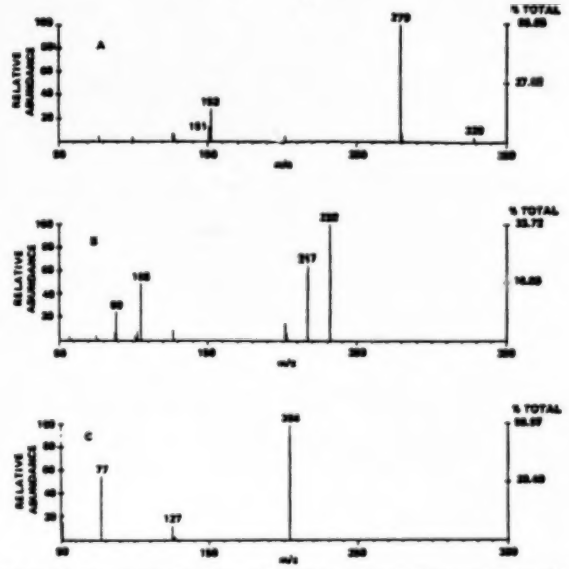

Figure 3. Mass spectra of HI derivatives of benzenearsonic acid (A), hydroxydimethylarsine oxide (B), and phenylmercuric acetate (C).

Table I. Summary of GC/FID and GC/MS Determinations of Candidate POHCs

	GC/FID		GC/MS	
compound	relative retention time[a]	on-column detection limit,[b] ng	relative retention time[a]	on-column detection limit,[b] ng
benzenearsonic acid	0.986	2.2	0.975	6.4
hydroxydimethylarsine oxide	0.324	11.8	0.301	5.7
phenylmercuric acetate	0.471	1.0	0.460	8.8
tetraethyllead	0.570	0.7	0.568	1.5
2,3,7,8-TCDD	1.26	0.7	1.23	0.8[c]

[a] Relative to the retention time of anthracene-d_{10}. [b] Quantity required to yield a detector response twice the magnitude of the background signal. [c] The detection limit by single ion monitoring is 0.1 ng.

two POHCs determined by HPLC/UV are presented. The high detection limit for benzenearsonic acid indicates that the HI derivatization method is the method of choice. Selenourea is easily determined by the HPLC/UV method presented in the experimental section.

Laboratory Recovery of Organometallics from Amberlite XAD-2 Resin, Glass-Fiber Filters, and Water. The sampling methods required for the ultimate analysis of POHCs in hazardous wastes and incinerator effluents during trial burns will vary according to the nature of the wastes. Prior to incineration, the hazardous waste may

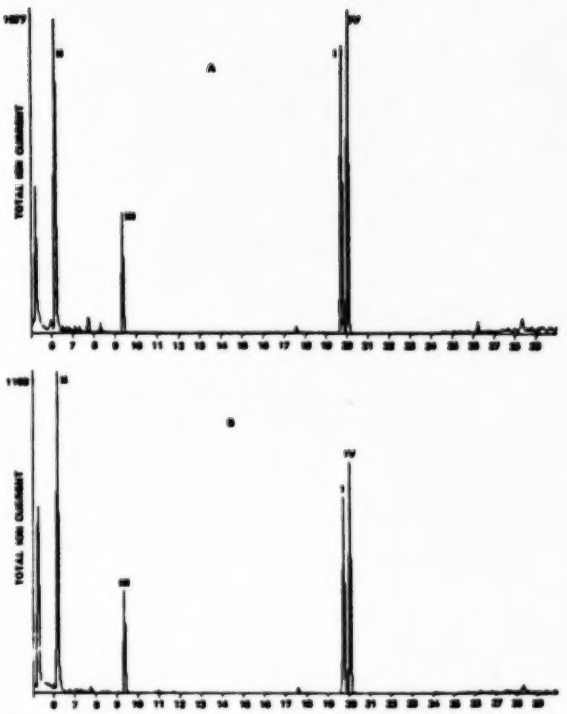

Figure 4. (A) GC/MS of standard containing the HI derivatives of benzenearsonic acid (I), hydroxymethylarsine oxide (II), and phenylmercuric acetate (III). Anthracene-d_{10} internal standard is IV. (B) GC/MS of the XAD-2 extract of the standard shown in (A).

be a solid, liquid, slurry, or sludge. After incineration, the POHCs may be found in bottom ash, fly ash/electrostatic precipitator (ESP) catches, scrubber water, or stack gas.

Sampling of the effluent streams of a hazardous-waste incinerator serves several purposes. During trial burns, the measurement of POHCs in the stack-gas effluent is an integral part of the calculation of DRE values to determine if the incinerator meets its performance criteria. The sampling of stack-gas components is important to the hazardous waste incinerator permitting process. Some of the sampling methods available to collect volatile POHCs at low concentrations in stack gas are the modified method 5 (MM5) sampling train, the source assessment sampling system (SASS), and the volatile organic sampling train (VOST). These systems use specific sorbents to collect POHCs.

XAD-2 resin, a general purpose sorbent, was selected for organic compounds to determine the recovery of POHCs from the sorbent. Glass-fiber filters and distilled deionized water were used to examine recovery from other parts of the sampling system. Methanol was used to extract benzenearsonic acid, hydroxydimethylarsine oxide,

Table II. Precision of Response Factors[a] for GC/FID and GC/MS Determinations

		GC/FID		GC/MS	
compound	M_r	mean (n = 3)	RSD, %	mean (n = 3)	RSD, %
benzenearsonic acid	202[b]	0.345	2	0.131	3
hydroxydimethylarsine oxide	138[c]	0.066	1	0.366	9
phenylmercuric acetate	337[d]	0.142	1	0.160	4
tetraethyllead	324	0.406	3	0.268	2
2,3,7,8-TCDD	320	0.041	4	0.048	3

[a] Response factor = (area std/area is)(concn is/concn std). [b] Molecular weight of HI derivative = 408. [c] Molecular weight of HI derivative = 232. [d] Molecular weight of HI derivative = 204.

Table III. Summary of HPLC/UV Determination of Candidate POHCs

compound	retention time, min	on-column detection limit,[a] μg	precision summary: quantity injected, μg	precision summary: mean area counts ×10⁻³	precision summary: RSD, %
selenourea[b]	3.5	0.0123	0.784	241	4.3
benzene-arsonic acid[c]	4.1	1.14	12.38	198	1.8

[a] Estimated quantity injected that is required to yield a response twice the magnitude of the background signal. [b] Wavelength of detection = 254 nm. [c] Wavelength of detection = 263 nm.

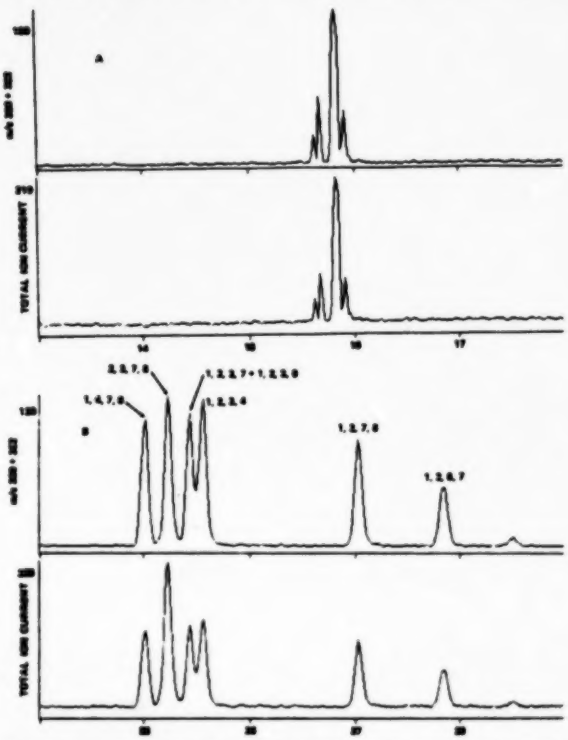

Figure 5. (A) Separation of seven TCDD isomers with the SE-54 GC column. (B) Separation of seven TCDD isomers with the CP-SIL-88 GC column.

and phenylmercuric acetate from the XAD-2 resins and glass-fiber filters. Water samples were evaporated to dryness prior to derivatization. Methylene chloride was used to extract tetraethyllead. The determination of the analytes was performed by GC/FID or GC/MS. Figure 2 is a diagram of one of the XAD-2 tubes used.

A representative GC/MS chromatogram of a standard containing HI derivatives of three organometallics is Figure 4A. Figure 4B is a GC/MS chromatogram of an extract of an XAD-2 tube that was exposed to moist air (≃85% humidity) at a flow rate of 200 mL/min for 2 h. The XAD-2/moist-air experiment was performed 6 times. One tube showed significant water condensation. The water was evaporated to dryness, and the residue was derivatized; the sum of the amounts found in the condensate and the XAD-2 resin was comparable to the other five determinations. Additional XAD-2 tubes were spiked and extracted after approximately 1 h and spiked and extracted after approximately 72 h. Table IV presents the recoveries of the three organometallics from the XAD-2 resin experiments. In a separate experiment, tetraethyllead was

Table IV. Recovery of POHCs from XAD-2 Resin and Glass-Fiber Filters

compound	XAD-2 resin extracted after 1 h: amount added, μg	XAD-2 resin extracted after 1 h: av % recovery (n = 3)	XAD-2 resin extracted after 1 h: % RSD	XAD-2 resin extracted after 72 h: amount added, μg	XAD-2 resin extracted after 72 h: av % recovery (n = 3)	XAD-2 resin extracted after 72 h: % RSD	XAD-2 resin extracted after moist-air exposure: amount added, μg lot 1	XAD-2 resin extracted after moist-air exposure: amount added, μg lot 2	XAD-2 resin extracted after moist-air exposure: av % recovery (n = 6)	XAD-2 resin extracted after moist-air exposure: % RSD	glass-fiber filter extracted after 72 h: amount added, μg	glass-fiber filter extracted after 72 h: av % recovery (n = 3)	glass-fiber filter extracted after 72 h: % RSD
benzene-arsonic acid	252	100	2.4	292	84	7.0	245[a,b]	315[b]	104	10	252	114	3.2
hydroxy-dimethyl-arsine oxide	278	92	7.8	278	69	8.7	300[b]	318[b]	82	16	278	99	4.2
phenylmercuric acetate	222	84	6.3	222	65	3.1	262[b]	300[b]	76	8	222	97	4.6
tetraethyllead[c]							248		91[d]	3			

[a] One sample had significant water breakthrough. Combinations of the derivatives of the liquid and XAD-2 extracts were analyzed. [b] Two sets of three samples were analyzed. [c] Only the moist-air exposure experiment was performed. [d] n = 3.

spiked onto XAD-2 tubes, exposed to moist air, and extracted with methylene chloride. Table IV also presents the data from the tetraethyllead tubes. The three organometallics were also spiked onto glass-fiber filters, extracted with methanol, derivatized, and analyzed by GC/FID. The recovery data are presented in Table IV for three spiked filters.

Because the three compounds were known to be somewhat water soluble, the compounds were spiked into 50 mL of distilled deionized water. Two experiments were performed. First, HI was added to the spiked water, the water was extracted with hexane, and then the extract was analyzed by GC/FID. Recoveries of hydroxydimethylarsine oxide and benzenearsonic acid were less than 3% and of phenylmercuric acetate greater than 100%. The recoveries from water may be influenced by the solubilities of these compounds in water and hexane. Second, the compounds were spiked into 50 mL of distilled deionized water, evaporated to dryness, derivatized, and dissolved in hexane, and the hexane was analyzed by GC/FID. The recovery of hydroxydimethylarsine oxide was 68%, of benzenearsonic acid was 87%, and of phenylmercuric acetate was 83%.

2,3,7,8-Tetrachlorodibenzo-*p*-dioxin. The determination of 2,3,7,8-TCDD by HRGC/LRMS was performed by using partial-scan MS (m/z 41–450 in 1 s) and selected ion monitoring (SIM) (m/z 257, 320, 322, 328, 332, and 334). The fused-silica bonded-phase SE-54 column was compared with the 2,3,7,8-TCDD isomer-specific fused-silica CP-SIL-88 column. The peak obtained for 2,3,7,8-TCDD with the more polar CP-SIL-88 column is somewhat broader than the peak from the SE-54 column, but the more polar CP-SIL-88 GC column provides separation of the 2,3,7,8-TCDD isomer from other interfering TCDD isomers. Figure 5A demonstrates the separation of seven TCDD isomers by using the SE-54 column, and Figure 5B is the separation of the same seven isomers by using the CP-SIL-88 column. Although the SE-54 column provides somewhat faster analysis times, the CP-SIL-88 column yields the isomer specificity necessary to perform the analysis. The detection of 2,3,7,8-TCDD at levels approaching 0.1 ng injected on the GC column (CP-SIL-88) has been estimated for reasonably clean samples. A sample containing 0.03 ng/μL 2,3,7,8-TCDD would give a signal detectable above background.

The screening methods evaluated for 2,3,7,8-TCDD were adapted from previously developed methods for specific POHCs (*6*). The purpose of this study was to evaluate how well the method would work for dioxins and furans if one simply "looked for them" during an analysis that was primarily for another POHC. Specific methods developed by Harless et al. (*9*, *10*), Karasek et al. (*11*, *12*), and Longbottom and Lichtenberg (14) may be more appropriate when only low levels of dioxins and furans are of interest.

Conclusions

A simple derivatization technique allows the determination of benzenearsonic acid, hydroxydimethylarsine oxide, and phenylmercuric acetate by the general GC/FID and GC/MS procedures. These compounds can be quantitatively recovered from materials used in stack sampling. The derivatization procedure may be useful for other organometallic compounds. Compounds such as selenourea and tetraethyllead may be determined by the HPLC/UV and GC/FID or GC/MS procedures, respectively, without major modifications of the procedures. The determination of 2,3,7,8-TCDD by the general procedures provides a screening analysis and would be readily applicable during the survey-analysis portion of hazardous-waste characterization. However, ultimate selectivity and sensitivity are provided by an isomer-specific GC column and selected-ion-monitoring mass spectrometry.

The determination of organometallics in field samples would aid in validating the method presented in this paper. During the incineration of many hazardous compounds, other compounds not already in the waste could be formed. For example, polychlorinated biphenyls can form polychlorinated dibenzofurans, or organometallic compounds may form inorganic oxides. The identification and development of analytical methods for these products of incomplete combustion (PIC) are under way.

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Received for review August 19, 1985. Revised manuscript received December 9, 1985. Accepted January 2, 1986. This paper has been produced by EPA's Office of Research and Development as part of on-going studies in support of regulatory programs and of EPA's Office of Solid Waste, EPA Regional Offices, and appropriate state agencies. The document contains state-of-the-art analysis methods for determination of performance of hazardous waste incinerators. This paper covers the laboratory phase of the study and does not include a discussion of actual combustion samples. It is intended as a reference work to be used by personnel of the regulatory groups, personnel associated with engineering R&D, and the regulated community. Inclusion in this paper does not mean that the sampling or analysis method is an official EPA method. The information in this document has been funded wholly by the U.S. EPA under Contract 68-02-3696. It has been subjected to the Agency's peer and administrative review, and it has been approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.